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DIELECTRIC PROPERTIES OF POLYMER MATRIX COMPOSITES PREPARED FROM CONDUCTIVE POLYMER TREATED FABRICS

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ABSTRACT

The dielectric functions of polypyrrole-treated polyester fabric and polypyrrole-treated S-glass fabric in polyester resin matrix composites have been calculated from free-space reflectance data in the 26.5 GHz to 40 GHz range. The data indicate that for the polypyrrole-treated polyester fabric/polyester resin composites, the imaginary part of the dielectric function can be described by a conductivity over frequency relationship. On the other hand, the real part of the dielectric function for these composites changes much less than the imaginary part when the conductivity of the fabric is increased. Similar results were found for the polypyrrole-treated S-glass in polyester resin composites. However, the real part of the dielectric function for these composites increases faster than for the polyester fabric composites. Scanning electron microscopy (SEM) indicated that the difference in behavior between the treated polyester/polyester and the treated S-glass/polyester composites can be due in part to nonuniform coating of the glass fabric. In contrast, the real part of the dielectric functions of several composites fabricated using S-glass woven roving with more uniform coatings were no larger than composites of the untreated rovings.

CONTENTS

INTRODUCTION

Conductive polymer treated fabrics and their composites are of great interest for their potential applications in electronic systems ranging from charge dissipation elements to shielding materials. Knowledge of the dielectric properties of conductive polymer-treated fabric polymer matrix composites is essential to design of these materials for electronic and/or microwave applications.

Composites of conductive polymer (CP) coated fabrics were expected to behave as essentially continuous conducting media, as opposed to granular media with discrete conducting particles such as carbon. It was anticipated that the dielectric behavior of the CP composites may be significantly different from that of granular composites in that it may be possible to introduce substantial dielectric loss without significantly altering the real part of the dielectric function.

This report presents results for the dielectric functions of composites of polypyrrole-treated polyester and polypyrrole-treated S-glass in polyester resin obtained from free-space reflectance measurements in the K_A band (26.5 GHz to 40 GHz). A series of samples were fabricated from treated polyester and S-glass fabrics (with various DC conductivities) and were examined. Measurements of composites of several weaves of treated S-glass were also taken. Scanning electron microscopy (SEM) was performed on some of the samples to aid in the interpretation of the results of the microwave measurements.

EXPERIMENTAL

The samples were prepared from fabric treated with polypyrrole at Milliken Research Corporation using a process which encases each individual fiber of the textile assembly with a smooth, nominally uniform, adherent layer of conductive polymer.[1] The polymerization of the textiles takes place at the surface of the fibers by controlling the reaction parameters such as monomer concentration, pH, and oxidizing agent. The reaction for the polymerization process commonly uses organic sulfonic acids as doping agents and the rate of reaction is controlled by introducing sulfosalicylic acid. The conductivity of the textile is varied by changing the amount of monomer available for the treatment of one particular batch of fabric. Aging, due to humidity, can have an effect on the electrical properties of the fabric and, as will be seen, can reduce the conductivity of the resultant composite.

The treated fabrics include the following:

(1) Milliken style 205 polyester fabric, a textured balanced twill weave with the same number of picks and ends suitable for clothing use. The doping agent used was 1,5-napthalene sulfonic acid.

(2) JPS S2-glass fabric style 016781, an 8.8 oz. basket weave, 55 picks by 57 ends with a JPS 09827 finish. The doping agent used was anthraquinone-2 sulfonic acid.

(3) A 5 x 5 S2-glass 24 oz. woven roving with an Owens Corning 463 finish. The doping agent used was 1,5-napthalene disulfonic acid.

1. GREGORY, R. V., KIMBRELL, W. C., and KUHN, H. H. Synth. Met., v. 28, C-823, 1989.

(4) A 3 x·1 S2-glass 27 oz. woven roving with an Owens Corning 933 finish, nominally equivalent to the JPS 09827 finish. The doping agent used was anthraquinone-2 sulfonic acid.

Only one conductivity was available for the fabrics listed in 3 and 4 above.

Composites of the polypyrrole-treated textiles were fabricated by laminating the layers of fabric with wet polyester resin (Owens Corning E-780) and subsequently processing the composites using the standard vacuum bag cure cycle for polyester-based material. The required thicknesses of the composites were determined by the desired appearance of structure (produced by multiple quarter-wavelength or half-wavelength interference patterns) in the measured reflectance.

The free-space reflectance apparatus used in this experiment is comprised of a Hewlett-Packard back wave oscillator source (HP 8690B) and a scalar network analyzer (HP 8757A) along with an attenuator, dielectric lens (or focusing horn), and a receiving antenna mounted on an X-ray defractometer turntable. The reflected amplitude was measured at angles of incidence of 16° and 45° as well as at a polarization parallel to the plane of incidence. At each angle, an open circuit (sample without a metal backing) and a short circuit (sample with metal backing) measurement was made.

The resultant data were analyzed by assuming that the radiation was a plane wave incident on a bulk slab of material at an arbitrary angle of incidence. The electromagnetic fields at the front surface of a material can be related to those at the back surface of the same slab of material by the expression[2,3]

$$\begin{bmatrix} E_1 \\ H_1 \end{bmatrix} = \begin{bmatrix} \cos(kd) & j\eta\sin(kd) \\ \frac{j\sin(kd)}{\eta} & \cos(kd) \end{bmatrix} \cdot \begin{bmatrix} E_2 \\ H_2 \end{bmatrix} , \tag{1}$$

or

$$\begin{bmatrix} E_1 \\ H_1 \end{bmatrix} = C \begin{bmatrix} E_2 \\ H_2 \end{bmatrix} . \tag{2}$$

From the above formulation for the 2 x 2 matrix C, $c_{11} = c_{22}$ and $c_{21} = c_{12}/\eta^2$. Also, as shown in Equation 1, E and H are the electric and magnetic fields, d is the thickness of the slab, k is the propagation constant, and η is the input impedance. For parallel polarization $\eta = z \cos \theta$ where $Z = \varepsilon/\mu$ and ε and μ are the dielectric and permeability functions (which are in general complex quantities). Also,

$$\cos\theta = \sqrt{1 - \frac{\sin^2\theta_i}{\varepsilon\mu}} , \tag{3}$$

2. CORNBLEET, S. *Microwave Optics*. Academic Press, Inc., New York, NY, 1976, p. 160-185.
3. JONES, D. S. *The Theory of Electromagnetism*. Pergamon Press, New York, NY, 1964, p. 316-329.

where θ_i is the angle of incidence. The propagation coefficient k is defined by the expression

$$k = \frac{2\pi f}{c} \sqrt{\mu\varepsilon} \cos\theta , \tag{4}$$

where f is the frequency of the incident radiation and c is the speed of light.

In general, the input impedance of the material is given by

$$Z_{\text{input}} = \frac{E_1}{E_2} = \frac{c_{11} Z_s + c_{12}}{c_{21} Z_s + c_{22}} , \tag{5}$$

where Z_s = the impedance of the sample. From Equation 5 the reflected power (db) is thus

$$R\,(\text{db}) = 20 \log_{10} \left| \frac{Z_{\text{input}} - \eta_o}{Z_{\text{input}} + \eta_o} \right| ,$$

where (6)

$$\eta_o = \sqrt{1 - \frac{\sin^2\theta_i}{\varepsilon\mu}} .$$

For the short circuit measurement (sample with a metal backing), Z_s = 0 and Equation 5 simplifies to

$$Z_{\text{input}} = \frac{c_{12}}{c_{22}} . \tag{7}$$

For the open circuit measurement $Z_s = \eta_o$, where the value of η_o was given in Equation 6. This is substituted into Equation 6 to obtain

$$Z_{\text{input}} = \frac{c_{11}\eta_o + c_{12}}{c_{21}\eta_o + c_{22}} . \tag{8}$$

The input impedance for both of the above cases can be substituted into Equation 6 to obtain the reflected power (db) for the desired fit to the measured data. The permeability μ was set to equal to 1 since the materials are nonmagnetic. Since no phase data were available, it was necessary to fit the observed data using the above expressions for the reflected power. An initial estimate of ε' (the real part of the dielectric function) was obtained from the position of the quarter-wavelength minima and an initial value of σ, the conductivity was obtained from the DC value and related to ε'' (the imaginary part of the dielectric function) by $\varepsilon'' = (36\pi \times 10^{11} \sigma)/(2\pi f)$. An iterative procedure was then used to obtain a value of $\varepsilon = \varepsilon' - i\varepsilon''$ which fit the data satisfactorily at both 16^o and 45^o. The procedure assumes ε' and σ are constant over the frequency range. This proved to be consistent with the data, although the sensitivity of the data to variation in ε' is small for the highest conductivity values. The values for the uncertainty in the reported results represent the change in the parameter needed to make a noticeable difference in the fit to the data.

RESULTS AND DISCUSSION

Polyester Fabric Composites

Figures 1a and 1b show, respectively, the measured reflectance and the fit generated using the above described formulation, for the open circuit case at angles of incidence of 16° and 45°, of the untreated polyester in polyester resin matrix composite, thickness = 0.952 cm. The best fit to the data was obtained by assuming $\varepsilon' = 2.65$, $\varepsilon'' = 0.05$, and $\sigma = 0$. The position of the minima and maxima and the magnitude of the reflected power determine the value for the fitted parameters. In this case, the fabric was not conductive and, therefore, the incident beam penetrated throughout the entire sample and multiple half-wavelength interference is observed. Figures 2a and 2b show the measured reflected power and the subsequent best fit to the data (for the open and short circuit measurement) at an angle of incidence of 16°, of the polypyrrole treated, 5000 ohms/square DC resistance, polyester resin matrix composite. The best fit was obtained by assuming $\varepsilon' = 2.7$ and $\sigma = 0.004$.

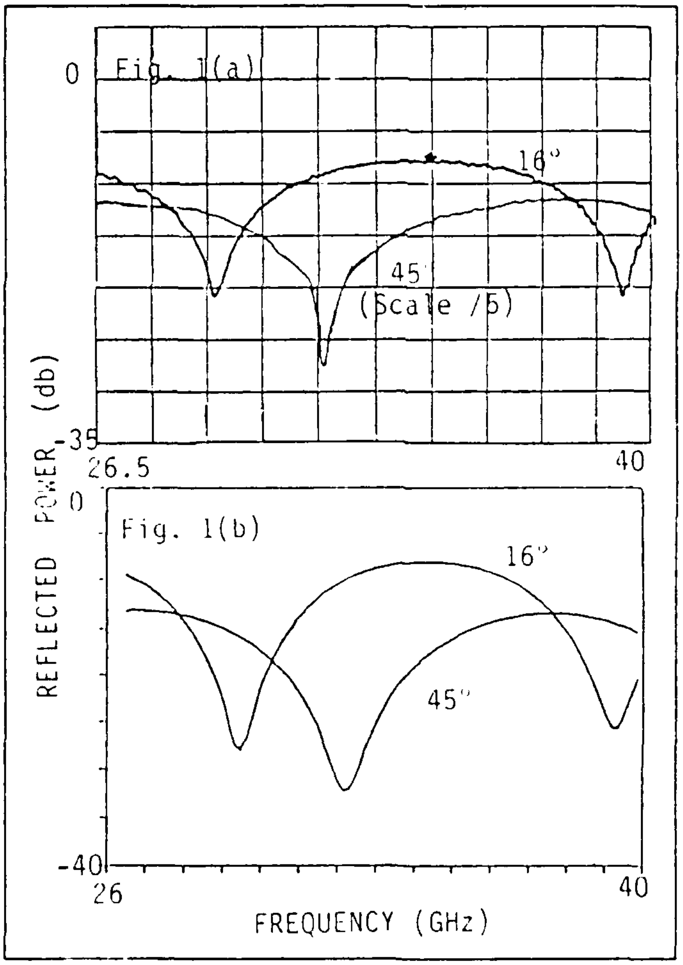


Figure 1. (a) The measured reflectance from untreated polyester/polyester composite from 26.5 GHz to 40 GHz, (b) the best to the above data using $\varepsilon' = 2.65$ and $\varepsilon'' = 0.05$.

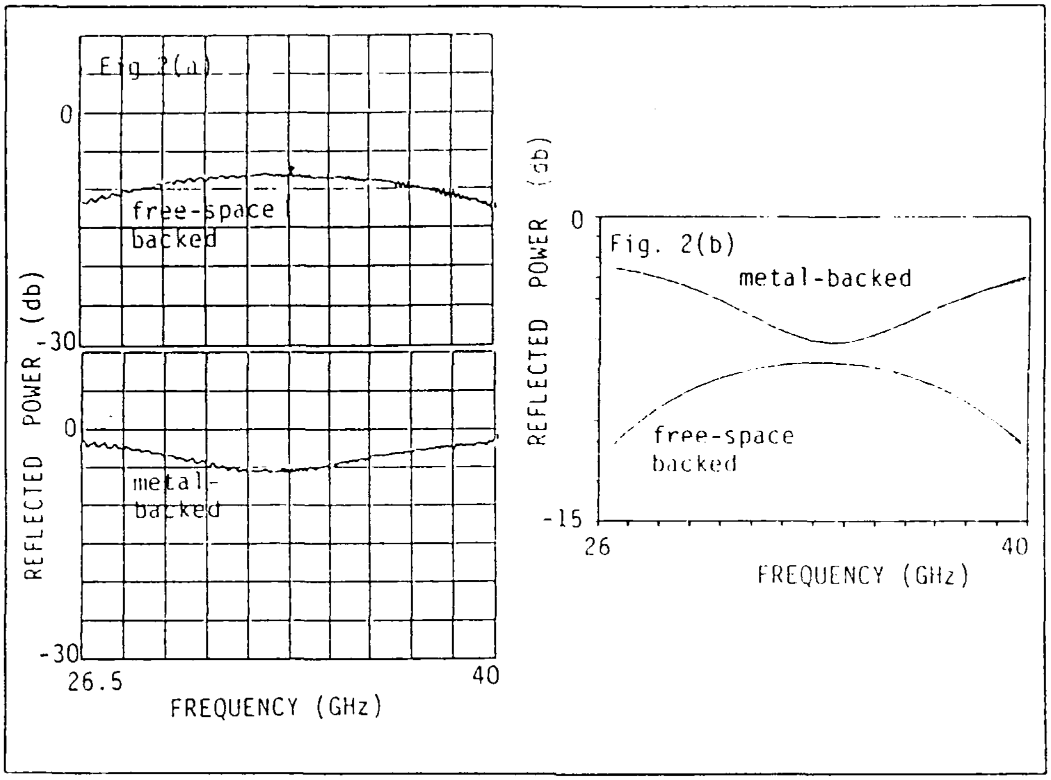


Figure 2. (a) The measured reflectance, open and short circuit, from a poly-pyrrole-treated, 5000 ohms/square DC resistance, polyester resin composite at $\theta_i = 16°$, (b) the best fit to the measured data assuming $\varepsilon' = 2.7$ and $\sigma = 0.004$.

The fitted parameters for ε' and σ, nominal DC resistance, and the sample thicknesses for all of the polypyrrole polyester in polyester resin composites are given in Table 1.

Table 1. BEST FIT PARAMETERS, ε' AND σ, OBTAINED FROM THE MEASURED REFLECTANCE IN THE 26.5 GHz TO 40 GHz REGION, OF THE POLYPYRROLE-TREATED POLYESTER IN POLYESTER RESIN MATRIX COMPOSITE

DC Resistance (ohms/square)	ε'	σ (S/cm)	Sample Thickness (cm)
---	2.65 ± 0.003	0.00 ± 0.0005	0.952
5000	2.70 ± 0.050	0.004 ± 0.001	0.419
2190	2.80 ± 0.050	0.012 ± 0.005	0.957
750	2.80 ± 0.100	0.030 ± 0.005	0.226
400	4.20 ± 0.200 (3.10*)	0.060 ± 0.005 (0.030*)	0.203, 0.152 (0.226*)
290	4.00 ± 0.200	0.060 ± 0.005	0.152
180	4.50 ± 0.200	0.090 ± 0.005	0.152
70	5.5 ± 0.500	0.150 ± 0.005	0.152
40	6.0 ± 0.500	0.450 ± 0.008	0.170

*Indicates a sample fabricated from a textile exposed to room humidity for several months.

Figures 3a and 3b show, respectively, ε' versus σ and ε' versus ε'' at 35 GHz. For lower values of σ, the relationship is linear and for σ up to 0.03 ε' changes very little from the value for the base composite (2.8 as compared to 2.65). For higher conductivities the real part of the dielectric function continues to increase and becomes nonlinear for $\sigma > 0.09$. In Figure 4, ε'' versus frequency is shown. This plot was obtained by using the conductivity over the frequency relationship mentioned above.

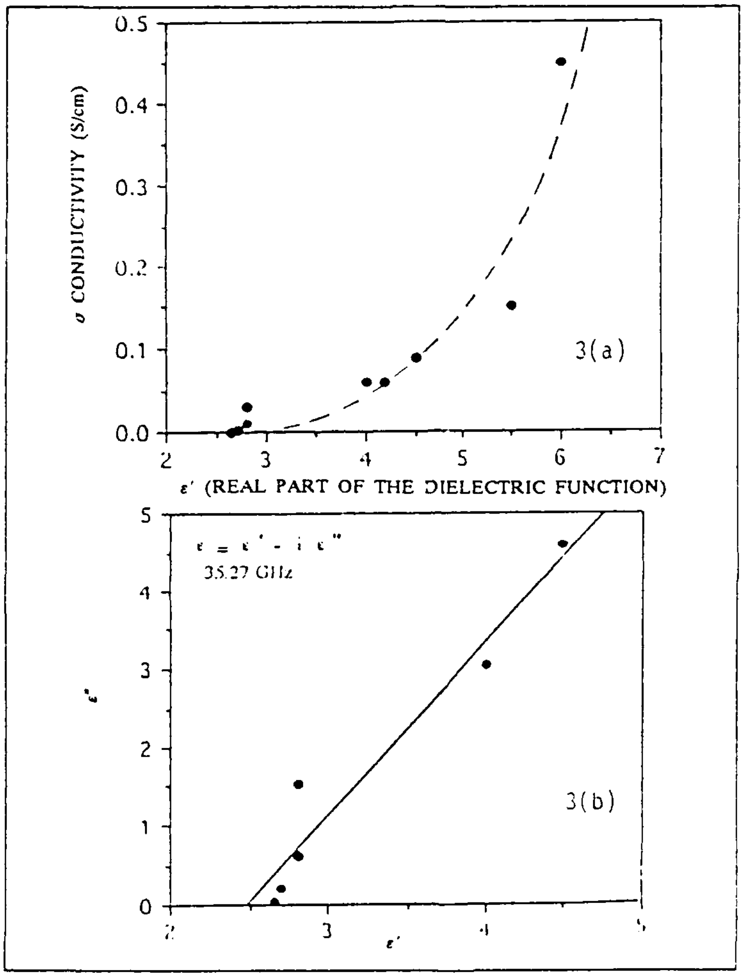


Figure 3. (a) ε' versus σ for polypyrrole-treated polyester in polyester resin matrix composites, (b) ε' versus ε'' for polypyrrole-treated polyester in polyester resin matrix composites.

As shown in Table 1, the effect of humidity on the treated fabric is shown by comparing the results for the fitted data for a composite fabricated with fabric which had been exposed to room environment for several months to a composite made from newly treated textile. As was expected, the fabric which was exposed for a long duration to room humidity has a much lower conductivity, $\sigma = 0.03$, as compared to the newer fabric, $\sigma = 0.06$. Aging of the DC electrical properties of the treated fabrics has been fully explored in a previous publication.[1]

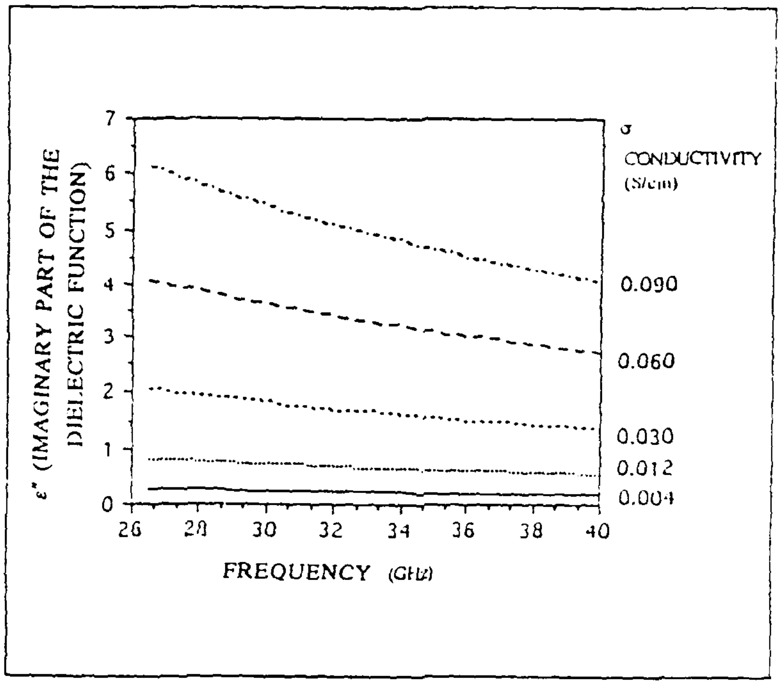


Figure 4. ε'' versus frequency, as obtained from a conductivity over frequency relationship, for the polypyrrole-treated polyester in polyester resin matrix composites.

S-glass Fabric Composites

The results for the untreated 8.8 oz. S-glass polyester resin matrix composite are shown in Figures 5a and 5b. In Figure 5a, the measured reflectance for the open circuit (without metal backing) at angles of incidence of 16° and 45° are shown. The best fit to the data in Figure 5a, assuming $\varepsilon' = 4.05$, $\varepsilon'' = 0.05$, $\sigma = 0$, and sample thickness = 0.635 cm, is shown in Figure 5b. As for the untreated polyester/polyester composite, the incident radiation penetrates through the entire sample and half-wavelength interference patterns are observed. An 8.8 oz. five harness satin weave rather than the basket weave fabric was used for this sample since none of the latter was available. The observed value is comparable to that obtained with other S-glass composites of comparable fiber volume.

The results for the measured and fitted data for the polypyrrole-treated S-glass sample, DC resistance 689 ohms/square, at angles of incidence of 16° and 45° are shown in Figures 6a and 6b, respectively. The best fit to the date was obtained with $\varepsilon' = 8.5$ and $\sigma = 0.100$.

A summary of the values obtained for ε' and σ along with the sample thickness and DC resistance for the polypyrrole-treated 8.8 oz. S-glass polyester resin matrix composites are shown in Table 2. In Figures 7a and 7b, ε' versus σ and ε' versus ε'' for the treated S-glass composites are shown. The behavior is linear even for the highest conductivity measured ($\sigma = 0.25$). In Figure 8, a plot of ε'' versus frequency has been obtained from a conductivity over frequency relationship. As was the case for the polyester/polyester composites, ε'' displays more metallic behavior with increasing conductivity.

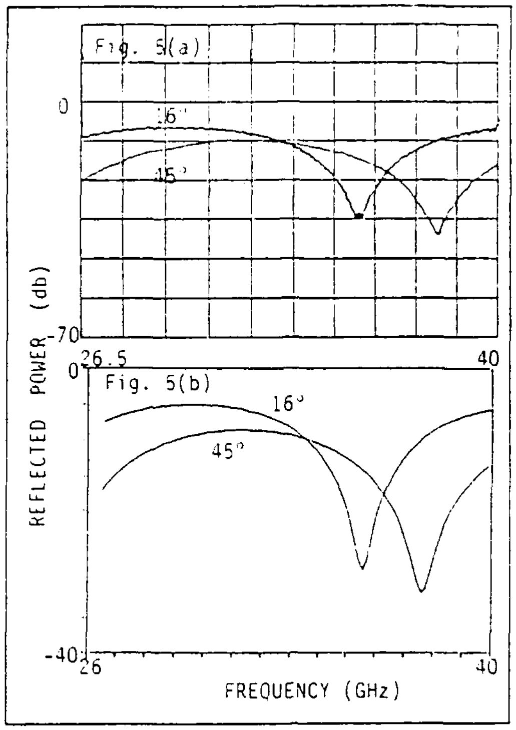


Figure 5. (a) The measured reflectance (open circuit) at θ_i = 16° and 45°, for the untreated S-glass polyester resin matrix composite, (b) the best fit to the data, assuming ε' = 4.05, ε'' = 0.05, and σ = 0.

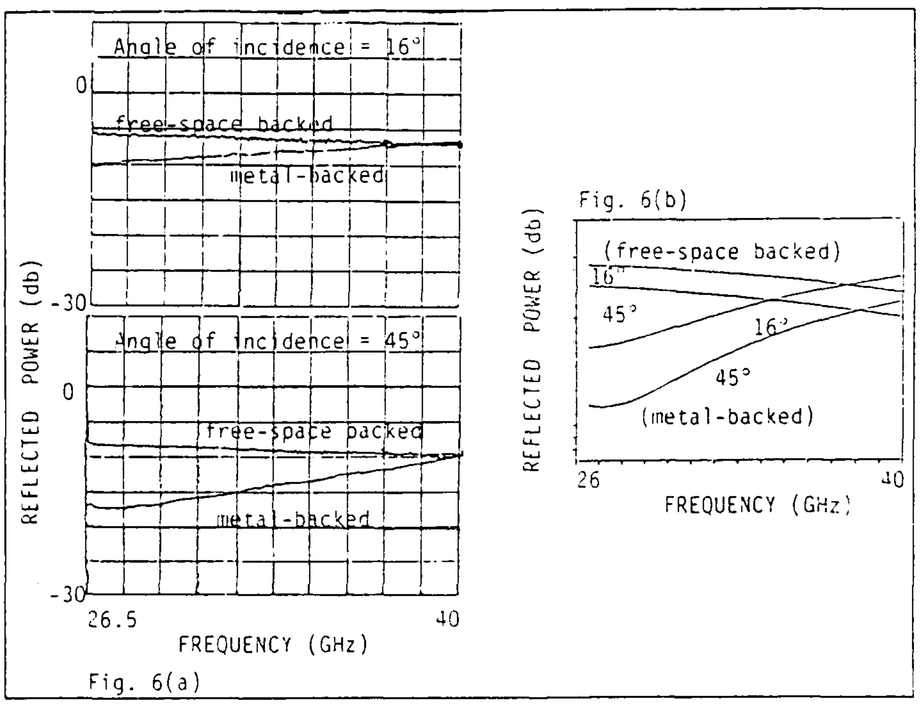


Figure 6. (a) The measured reflectance for the polypyrrole-treated S-glass, 689 ohms/square, polyester matrix composite at θ_i = 16° and 45°, (b) the best fit to the data in figure 6(a) assuming ε' = 8.5 and σ = 0.100.

Table 2. BEST FIT PARAMETERS, ε' AND σ, OBTAINED FROM THE MEASURED REFLECTANCE IN THE 26.5 GHz TO 40 GHz REGION, OF THE POLYPYRROLE-TREATED S-GLASS POLYESTER RESIN MATRIX COMPOSITE

DC Resistance (ohms/square)	ε'	σ (S/cm)	Sample Thickness (cm)
- - -	4.05 ± 0.005	0.00 ± 0.0005	0.635
226	7.00 ± 0.10	0.050 ± 0.005	0.102
1487	6.80 ± 0.050	0.080 ± 0.005	0.102
689	8.50 ± 0.100	0.100 ± 0.005	0.102
458	9.00 ± 0.200	0.110 ± 0.005	0.102
279	10.0 ± 0.500	0.200 ± 0.010	0.102
127	12.0 ± 0.500	0.250 ± 0.020	0.102

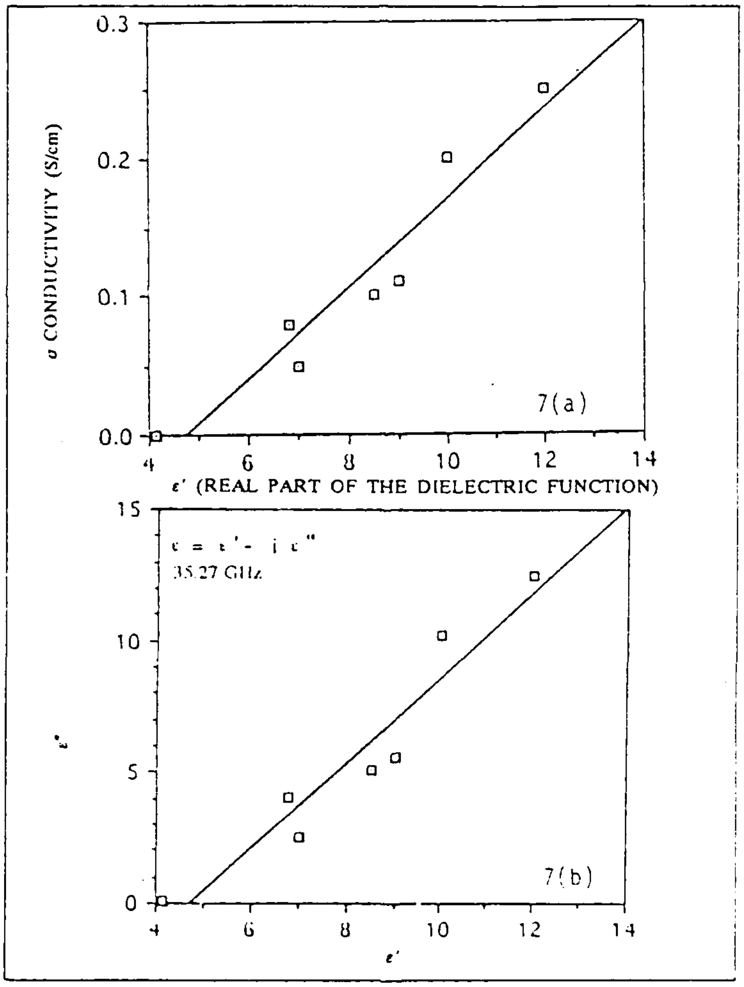


Figure 7. (a) ε' versus σ for polypyrrole-treated S-glass in polyester resin matrix composites, (b) ε' versus ε'' for polypyrrole-treated S-glass in polyester resin matrix composites.

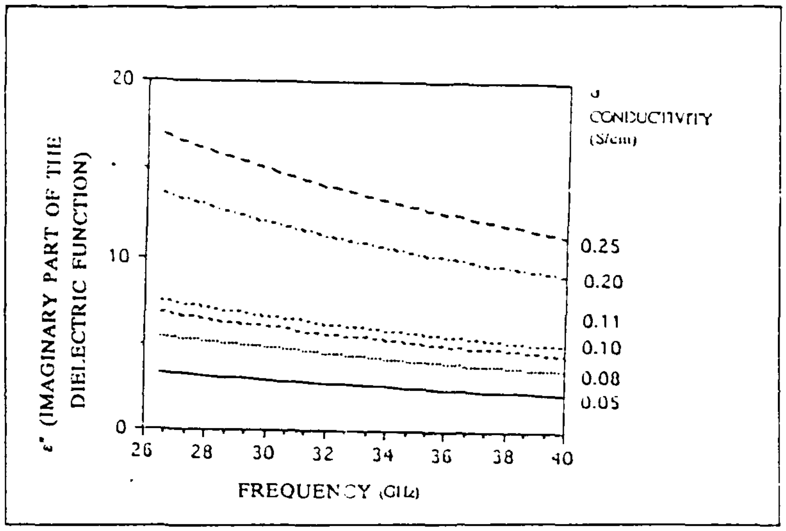


Figure 8. ε" versus frequency, as obtained from a conductivity over frequency relationship, for the polypyrrole-treated S-glass polyester resin matrix composites.

Two treated S-glass woven rovings were also examined. Only one conductivity for each was available, but the results are in sharp contrast to the 8.8 oz. materials. For the 5 x 5, 24 oz. roving, the base composite dielectric constant is 4.14 -i0.05, whereas the treated roving composite showed a real dielectric constant of only 3.4 and a conductivity of 0.09. The 3 x 1, 27 oz. roving based composite had a dielectric constant of 3.8 -i0.10, while the treated roving had a real dielectric constant of 3.8 and a conductivity of 0.05.

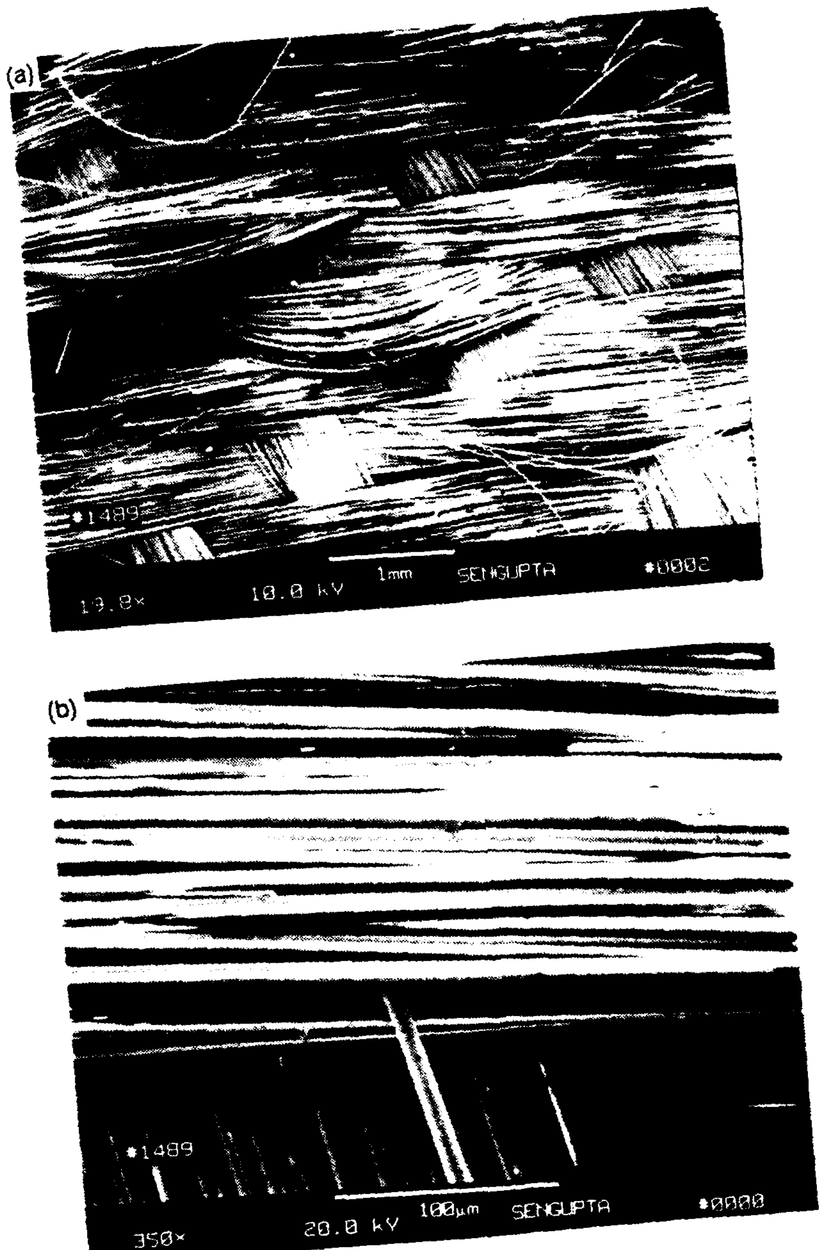
The increase in ε' with conductivity for the 8.8 oz. S-glass composites is more than proportionally larger than that of the polyester fabric composites and is larger than that of the two heavy roving S-glass composites. This suggests that the 8.8 oz. S-glass composites are somewhat more granular in nature; i.e., that the coating may not be continuous on the fabrics. SEM analysis of the coatings was, therefore, performed to try to validate this hypothesis.

SEM images of a polypyrrole-treated polyester fabric, 2190 ohms/square (DC measurement) and a polypyrrole-treated S-glass textile, 1487 ohms/square (DC measurement) are shown in Figures 9 and 10. The polyester fabric at both magnifications does not show any areas which are charging up (see Figures 9a and 9b) even at such a low conductivity. On the other hand, the treated S-glass (see Figures 10a and 10b) shows distinctive regions on the sample at both magnifications which are charging up due to a lack of or a much thinner conductive coating in these areas.

The 8.8 oz. S-glass fabrics were initially sized with the Owens Corning 463 sizing. Since this sizing is not optimum for the polypyrrole treatment, it was burned off and the fabric was subsequently cleaned in methylene chloride and resized with the JPS 09827 finish prior to polypyrrole coating. It is possible that the fabric was somewhat dirty on a microscopic scale as a consequence, which could well lead to uneven coating distribution. All SEM samples of this S-glass fabric showed the charging behavior from the evidently nonuniform coating. The 5 x 5 roving also looked uniform microscopically, although a few macroscopic patches of untreated fabric were noted.

Figure 9. (a) SEM of polypyrrole-treated polyester, 2190 ohms/square DC resistance, polyester resin matrix composite, magnification 26.8X, (b) SEM of polypyrrole-treated polyester, 2190 ohms/square DC resistance, polyester resin matrix composite, magnification 344X.

(a)



(b)

Figure 10. (a) SEM of polypyrrole-treated S-glass, 1489 ohms/square, DC resistance, magnification 19.8X, (b) SEM of polypyrrole-treated S-glass, 1489 ohms/square, DC resistance, magnification 350X.

CONCLUSION

The dielectric properties of polypyrrole-treated polyester fabric and S-glass fabric polyester resin matrix composites with various conductivities have been obtained from free-space reflectance data in the 26.5 GHz to 40 GHz range. The treated polyester/polyester composites show very little change in ε' as compared to the untreated material up to $\sigma = 0.09$ beyond which the dielectric behavior is nonlinear. The coating on the fibers used in these composites appears to be uniform from the SEM images that were obtained. On the other hand, the treated 8.8 oz. S-glass composites have dielectric behavior very different from the base composite, and this behavior is linear up to $\sigma = 0.25$. The coating on the fibers used in these composites is, apparently, not uniform as determined from SEM images. This nonuniformity, probably attributable to processing difficulties, may contribute to the more granular behavior of the electrical properties in this frequency range. Two different treated S-glass woven rovings were also examined. Composites of these rovings exhibited electrical behavior similar to that of the polyester fabric composites.

Studies of the dielectric behavior of these composites in other frequency bands are planned.

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DIELECTRIC PROPERTIES OF POLYMER MATRIX COMPOSITES PREPARED FROM CONDUCTIVE POLYMER TREATED FABRICS -
Louise C. Sengupta and William A. Spurgeon
Technical Report MTL TR 92-3, February 1992, 16 pp-illus-tables

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Key Words

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Microwave measurements
Polyester resins

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